

ORIGINAL RESEARCH PAPER

## Application of Granular Ferric Hydroxide in Removal of Nitrate from Aqueous Solutions

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**ABSTRACT:** The aim of this study is to investigate the nitrate adsorption on granular ferric hydroxide (GFH). The effect of factors such as initial concentration of nitrate, pH, contact time, and adsorbent mass were determined. The study was done in laboratory-scale in which synthetic nitrate solutions in a batch reactor were placed in contact with the adsorbent to model adsorption kinetics. It was found that with increasing initial concentration of nitrate, sorption capacity reaches the maximum level within two to three minutes. Increasing the amount of adsorbent and decreasing the adsorbent particle size could reduce the adsorption capacity. At alkaline pH (9.5), the highest sorption of nitrate on GFH occurred but it was found that the pH does not have a great impact on nitrate adsorption on GFH. Also, it was revealed that the adsorption kinetics followed pseudo-degree reaction model. This method could decrease the nitrate concentrations to less than the standard level (45 mg/L based on nitrate).

**Keywords:** Adsorption, Underground Water, Kinetics, Potable Water, GFH

### Introduction

In many parts of the world, people use groundwater sources as potable water. Thus, possible pollutants in these water resources should be considered. Recently, agricultural and industrial works have caused toxic pollutants in groundwater. These pollutants include inorganic anions such as nitrate and fluoride, arsenic, metal ions, and synthetic organic chemicals [1]. Nitrate can penetrate to water through air and soil. Excess nitrate in drinking water is a carcinogen and toxic for human and animals [2, 3].

Environmental agencies have defined various maximum contaminant level (MCL) for nitrate intake. EPA has established 10 mg/L of nitrate and WHO has determined 45 mg/L of nitrate as MCL for this ion [1, 4].

If nitrate concentration in potable water exceeds the standard level, it can cause environmental issues like etherification in rivers and illnesses such as methemoglobinemia and other health problems as blood pressure [5, 6], congenital defects including cardiac defects [7, 8], colon cancer [9], goiter, thyroid disorders, gastric cancer [10], increase in children mortality and genetic defects [7] in human.

There are various methods to remove nitrate from drinking water that could be classified as chemical (e.g. electrolysis [11–15], reverse osmosis [10, 16], ion exchange [10, 17], coagulation, electrical, and chemical coagulation [18]), physical (e.g. adsorption [1, 19]), physicochemical [20], biological [10] (e.g. denitrification [21]), and bioreactor membrane [22] groups.

Adsorption method has been removed diverse inorganic

anions such as fluoride, nitrate, bromate and perchlorate from water by different types of adsorbents [1].

Various adsorbents have been used for nitrate adsorption including PAC, ZnCl<sub>2</sub> treated coconut granular, bamboo charcoal, layered double hydroxide etc. [1]. Granular ferric hydroxide (GFH) is a substance that has been used as adsorbent for removing some of pollutants including six-valent chromium [23], NOM [24], fluoride [25] and arsenate [26].

Due to GFH is an environment-friendly substance [25] and its costs are lower than other removal methods, researchers are encouraged to use this substance as an adsorbent.

The aim of this study is to investigate the nitrate adsorption on granular ferric hydroxide (GFH). The effects of factors such as initial pH, contact time, initial nitrate concentration, and adsorbent mass were also determined.

### Materials and methods

This is an original study and it was done in laboratory and pilot scale. The stages of preparation and experiment are offered as bellow:

#### Granular ferric hydroxide (GFH)

GFH was purchased from Wasserchemie (GmbH & Co. KG, Osnabruck, Germany). The granules were sieved by 18 mesh sieve until granules smaller than 1mm got completely separated. Table 1 demonstrates GFH features. The specific surface area of GFH was determined through BET method. A total of 97% of the pores is less than 4.5 nm [5]. The pH of the point of zero charge (pH<sub>PZC</sub>) was determined by different researches to be 7.5 – 8 [5]. Then, GFH was washed with deionized water and placed in an oven for 2 h at 105 °C temperature.

#### Nitrate solution preparation

Nitrate stock solution (1000 mg/L of nitrate) was prepared

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by dissolving  $\text{KNO}_3$  (Merk Co, Germany) in de-ionized water. A range of dilutions (50 – 150 mg/L of nitrate) were prepared from the stock solution.

**Table 1.** Characteristics of GFH

Character	Value
Specific surface area ( $\text{m}^2/\text{g}$ )	280
Particle size (mm)	0.32 – 2
Water content (%)	43 – 48
$\text{pH}_{\text{PZC}}$	7.5 – 8.2
Bulk density ( $\text{kg}/\text{m}^3$ )	1250
Porosity of grains (%)	72 – 77

### Theory

At first, a pretest was done to specify the optimum adsorption pH and proper contact time limit. The nitrate concentration and contact time limit for pretest were selected 50 mg/L of nitrate and 5 – 90 min, respectively. To adjust pH about 4, 7, and 9.5 as acidic, neutral and alkaline pH, respectively,  $\text{H}_2\text{SO}_4$  and  $\text{NaOH}$  were used. It was observed that after 5 min contact time, desorption of nitrate happened. Therefore, the experiments were also done under 5 min and it was seen that nitrate adsorption on GFH granules occurs under 5 min contact time. Thus, in the following experiments, samplings were done less than 5 min in duration.

The adsorption experiments were done on GFH with a smaller diameter than 1 mm and initial nitrate solutions of 50, 75, 100, and 150 mg/L of nitrate and fixed volume of 50 mL to determine the optimum initial concentration of nitrate. Due to specify the optimum mass of adsorbent, the experiment was conducted with various masses of GFH including 0.625, 1.25, 2.5, and 3.75 g in 50 mL fixed volume of nitrate solution with the optimum initial concentration of nitrate, which was adopted from the previous stage. Finally, the adsorption process at the optimum condition, which was achieved from previous experiments, was investigated at 0.5, 1, 1.5, 2, 3, 4, and 5 min to find out the optimum contact time of adsorption. All experiments were studied at room temperature ( $25 \pm 2$  °C) and mixed with a shaker (300 rpm). After equilibrium, samples were filtered using 0.45  $\mu\text{m}$  membrane filters and the concentration of nitrate in residual solution was determined by DR4000 spectrophotometer (HACH) at 220 nm wavelength.

### Adsorption studies

Eq. 1 and Eq. 2 are used to determine the amount of adsorbed nitrate (mg) on one gram adsorbent.

$$q_e = \frac{(C_0 - C_t)V}{m} \quad (1)$$

$$q_t = \frac{(C_t) \times V}{m} \quad (2)$$

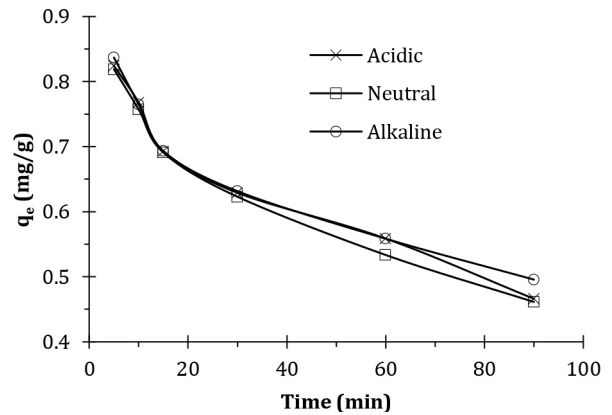
Where  $q_e$  is the amount of adsorbed nitrate on adsorbent at equilibrium (mg/g),  $q_t$  is the amount of adsorbed nitrate on adsorbent at t time (mg/g),  $C_0$  is the initial concentration of nitrate in solution (mg/L),  $C_t$  is the final concentration of nitrate in solution (mg/L),  $m$  is the adsorbent mass (g), and  $V$  is the solution volume (L).

The obtained results from this study were plotted and described by using Excel software and Squared value (R).

## Results and Discussion

### Effect of pH

The effect of acidic, neutral and alkaline pH (4, 7, and 9.5, respectively) on adsorbing nitrate on GFH at a specified period of contact time (5 – 90 min) with an initial concentration of 50 mg/L of nitrate was shown in Fig. 1.



**Fig. 1.** Effect of pH on adsorption of nitrate on GFH (temperature= 25 °C, initial nitrate concentration= 50 mg/L of nitrate)

pH plays a remarkable role in adsorption treatment processes of water because proton concentration can strongly change the redox potential of adsorbates and adsorbents, enhance dissolution of the adsorbent material, and modify chemical speciation of the adsorbates as well as a surface change of adsorbent [27]. The effect of pH on nitrate adsorption on GFH was studied by a pH range of 4, 7, and 9.5 as acidic, neutral and alkaline pH, respectively. Notice to Fig. 1, it could be said that differing pH (acidic, natural, and alkaline) does not have a significant impact on removing nitrate by GFH, but alkaline pH is better than acidic and neutral pH. Thus, for experiment accuracy, the alkaline pH was preferred as optimum pH for nitrate adsorption on GFH.

Tang showed that the maximum adsorption of fluoride happens at 3 – 7.5 pH limit [25]. Asgari found out that the highest arsenic [28], arsenate, arsenite and chromate [23] adsorption on GFH occurs at neutral pH (about 7). Oztork observed that the effective pH for removing nitrate by PAC is equal to 2 and mentioned that the pH amount does not have a significant impact on nitrate adsorption on other adsorbents [29].

### Effect of initial nitrate concentration

In Fig. 2, the impact of initial concentration (50, 75, 100, and 150 mg/L of nitrate) at specified period of time (0.5 – 5 min) was illustrated. Four different initial concentrations (50, 75, 100, and 150 mg/L) of nitrate was applied to study the effect of initial nitrate concentration on nitrate adsorption on GFH. Fig. 2 indicates that the required time to reach equilibrium based on initial concentration of nitrate is variable, 3 min ( $C_0 = 75, 50,$  and  $100$  mg/L) and 2 min ( $C_0 = 150$  mg/L). In addition, it was recognized that the nitrate removal potential by GFH at equilibrium concentration increased with increasing the initial concentration.

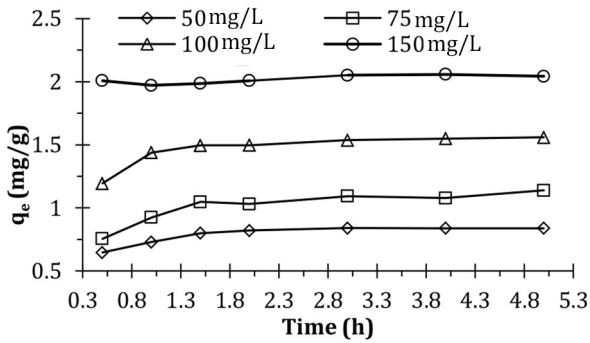


Fig. 2. Effect of initial nitrate concentration on nitrate adsorption on GFH (temperature= 25 °C, pH= 9.5)

Effect of adsorbent mass

In Fig. 3, the effect of adsorbent mass on adsorption kinetic by time could be seen. There is a relation among the number of active sites and adsorbent mass. The adsorbent mass due to having adsorption active sites can play an important role in removing pollutants from aqueous solutions. Thus, the effect of GFH mass on nitrate adsorption was investigated at four various mass of GFH (0.625, 1.25, 2.5, and 3.75 g). Fig. 3 indicates that by increasing the amount of adsorbent (0.625 – 3.75 g), nitrate adsorption capacity decreases. The maximum value of equilibrium concentration is belong to minimum GFH mass (0.625 g), which reaches an equilibrium state after 3 min. The minimum equilibrium concentration belongs to the maximum adsorbent mass (3.75 g), which after 1 min gets to equilibrium states.

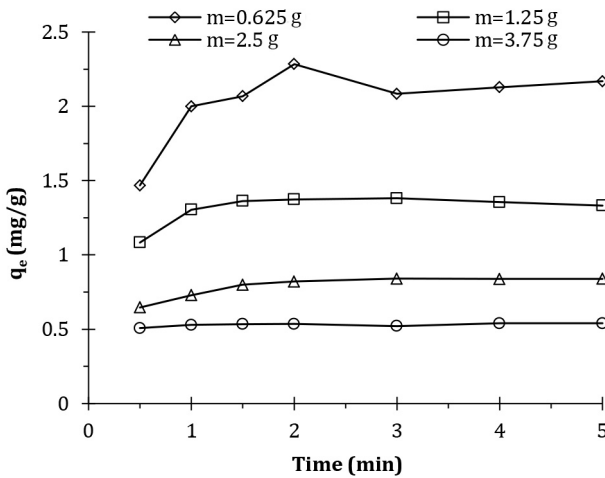


Fig. 3. Effect of GFH mass on nitrate adsorption on GFH (temperature=25 °C, pH= 9.5, initial nitrate concentration= 50 mg/L of nitrate).

Effect of contact time

The effect of contact time on adsorbing nitrate by GFH was studied. It was observed that the best removal of nitrate happens at the first 5 min of the contact time. During the pretest, the contact time effect was examined between 5 min and 90 min. It was observed that the maximum removal of nitrate happens during the first 5 min and after 5 min due to completion of GFH’s adsorption capacity, disadsorption occurs. Thus, the next experiments were done under 5 min contact time. Fig. 4 demonstrates the adsorption kinetics of nitrate uptake by GFH. As shown in Fig. 4, it could be found out that the adsorption of nitrate happens within the first few minutes of contact time between aqueous and adsorbent, and then it

rises till reaches equilibration status. It could be said that removing nitrate ions take place in short time and the required time for equilibration is about 2 to 3 minutes and after 3 min there is no significant adsorption. These observations are similar to what Chabani found out [30].

Bhatnagar observed that in removing bromate from water with GFH by increasing the time, bromate removal increased. He indicated that adsorbent exhibited an initial rapid uptake removing nearly 75% of the dissolved bromate within the first 5 min followed by a slow kinetics to reach a plateau. He selected 60 min as equilibration period [31].

Fluoride adsorption on GFH was increased by increasing time. 90% of fluoride was removed within the first 10 min and only 3 – 4% of removal happened within next 24 hours [32].

Kumar indicated that perchlorate adsorption on GFH increased by increasing time. The maximum removal of perchlorate occurred in the first 3 min and at 60 min reached the equilibrium [33].

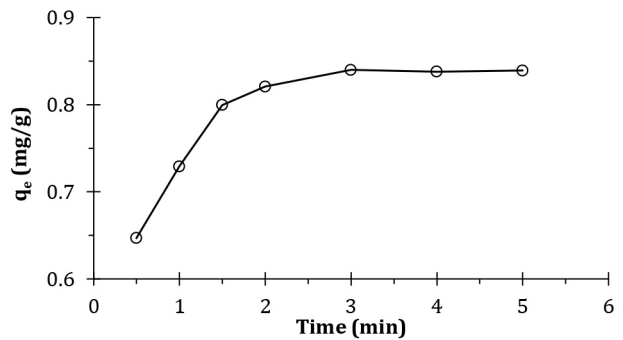


Fig. 4. Nitrate adsorption kinetics on GFH (temperature= 25 °C, pH= 9.5, initial nitrate concentration= 50 mg/L of nitrate, GFH mass= 0.625 g).

Kinetics modeling

Fig. 4 shows the nitrate adsorption kinetics and the results of Lagergren and pseudo second order kinetics studies are given in Fig. 5 and 6.

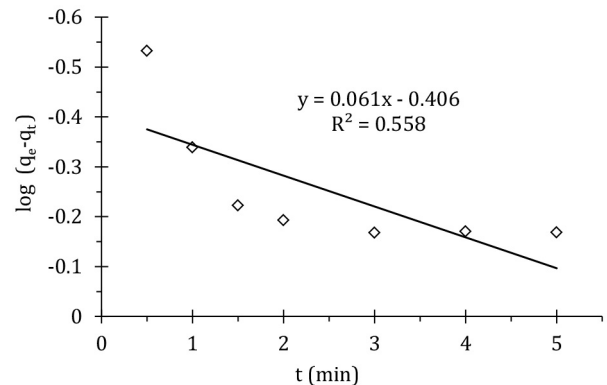


Fig. 5. Nitrate adsorption kinetic modeling on GFH, pseudo– first–order (Lagergren) (temperature= 25 °C, pH= 9.5, initial nitrate concentration= 50 mg/L of nitrate, GFH mass= 0.625 g).

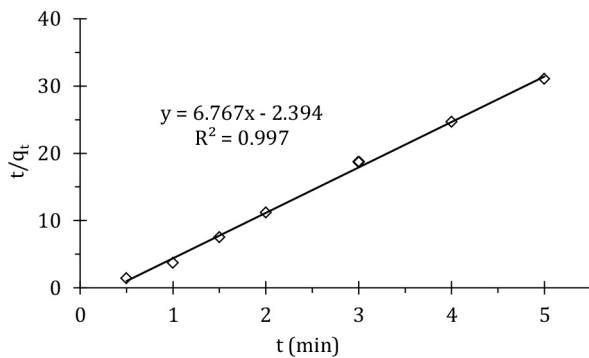


Fig. 6. Nitrate adsorption kinetic modeling on GFH, pseudo-second-order (temperature= 25 °C, pH= 9.5, initial nitrate concentration= 50 mg/L of nitrate, GFH mass= 0.625 g).

One of the more important characteristics in defining the efficiency of adsorption is kinetics modeling. There are various kinetics models which are proposed by different investigators. In the present study, two kinetics models including pseudo-second-order and pseudo-first-order model have been discussed to investigate the rate and mechanism of nitrate sorption on GFH.

#### Pseudo-first-order model

The Lagergren's rate equation is one of the most widely used rate equation which describes the adsorption of an adsorbate from the liquid phase. The linear form of pseudo-first-order rate expression of Lagergren is as below:

$$\log(q_e - q_t) = \log q_e - \frac{k_f}{2.303} t \quad (3)$$

Where,  $q_e$  and  $q_t$  have the same meaning as mentioned before.  $k_f$  is the rate constant of Lagergren (1/min) which is determined from the slope of plot and  $q_{e(\text{cal})}$  is obtained from the intercept of the plot [32].

#### Pseudo-second-order model

The adsorption kinetics can also be modeled by pseudo-second-order equation as following:

$$\frac{t}{q_t} = \frac{1}{k_s q_e^2} + \frac{1}{q_e} t \quad (4)$$

Where,  $q_e$  and  $q_t$  are as mentioned perversely.  $k_s$  is the rate constant for the pseudo-second-order kinetics (g/mg).  $q_e$  and  $k_s$  are determined from the slope and intercept of the plot, respectively [32].

As observed in Fig. 5 and 6, the  $R^2$  value for pseudo-second-order model is significantly greater than that for the Lagergren model. Nitrate adsorption on GFH follows the pseudo-second-order reaction model with  $R^2 = 0.977$ .

## Conclusions

The results indicated that GFH had a high efficiency in removing nitrate from potable water even at high concentrations of this pollutant. Due to pH does not have a noticeable effect on removal results of this pollutant, using neutral pH or in another word the natural pH of water, in functional scale without adjusting pH is possible. This can cue reduction in operational costs. Therefore, it is one of the benefits of employing GFH in removing nitrate.

GFH in water remains iron and can cause color in water but conventional water treatment plants can eliminate this residual iron. For utilizing this method for nitrate removal in large scale, it is suggested to use it before carbon active unit or sand filters. Also, it is better to localize GFH manufacturing technology to decrease the cost of purchasing it from abroad. One of the disadvantages of this method is the used GFH in the removal method which after employing contains nitrate and needs to be disposed of. If a method could proceed to reduce the used GFH and reuse it, the costs of this removal method would decrease and there would not furthermore request to dispose of the remained GFH.

In several studies, the effect of the temperature on the adsorption process has been proven, but due to the lack of lab facilities, the effect of temperature on the adsorption was not investigated in this study. It is suggested for further studies to investigate the effect of the temperature on the adsorption process.

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